

Experimental Systems Group Overview

Many of the projects outlined in this section are also described in more detail in the individual beamline sections of this compendium. The intention here is to give an overview of the range of projects carried out by the Experimental Systems Group and to highlight areas of special interest.

Beamline 1.4

Beamline 1.4 was originally designed to have a single endstation for infrared (IR) microscopy. This system uses a commercial IR spectromicroscope manufactured by Nicolet, and is connected to a bend magnet port via a plane premirror, an ellipsoidal mirror, and collimating optics. This microscope is now in routine use. A number of upgrades have been carried out during the year to reduce optical noise. The noise came from two sources: low-frequency noise induced into the optical mountings by floor vibration and high-frequency electron-beam noise. The latter problem was traced to a digital synthesizer used as the master ring oscillator, and has been largely eliminated by replacing the unit with a higher-quality unit with much lower sidebands. The problem of floor vibration has been traced to the rf water pumping system, and the noise has been reduced by replacement of the water pump with a variable-frequency type operating at reduced pressure. In addition, it was found that the premirror assembly was vibrating excessively, and this was traced to the separation of the upper and lower concrete floor slabs in the IR area. This was solved by separation of the system from the upper slab and direct bolting to the lower slab. In order to reduce vibration to a minimum, in the next year we will continue to reduce the vibrations by isolation of the rf water pump and by more rigidly mounting the external switchyard mirror system. This is planned for the June 1999 shutdown. In addition, one of the switchyard mirror mounts will be replaced with a piezo-driven system, and this will be used in an active feedback loop to further reduce the effect of low-frequency vibrations. The system is currently performing well and is in routine use by a wide range of users for research ranging from environmental science to structural biology. The beamline was modified during the year to extract two new lines, one for surface infrared reflection spectroscopy of surfaces (Ross) and the other for UV-visible spectroscopy of defects in semiconductors (Haller). These beamlines are essentially complete and are undergoing commissioning.

Martin, M.C., and W.R. McKinney, "The first synchrotron radiation infrared beamlines at the Advanced Light Source: microspectroscopy and fast timing," in *1998 Spring Meeting of the Materials Research Society*, Proceedings of Materials Research Society **524**, 11, April 12-16, 1998, San Francisco, CA, USA.

Beamline 3.3

The deep lithography LIGA system was moved from Beamline 7.3.3 in January 1998 to its new location on Beamline 3.3. It was operated for the first time just before the April 1998 shutdown. The LIGA consortium includes groups from Sandia National Laboratories, the Jet Propulsion Laboratory, and Lawrence Berkeley National Laboratory. The work centers around the manufacture of various types of high-aspect-ratio structures, from parts for micromachines to x-ray telescope collimators. The scanner itself was reengineered to allow substrate cooling, and many modifications were made to provide more reliable operation. The LIGA scanner simply oscillates the sample, a photoresist-coated wafer, in the white x-ray beam to achieve a uniform exposure. The system is controlled to give precise radiation doses and is nitrogen-flowed to exclude oxygen and minimize ozone production. The beamline is simply an evacuated tube, terminated in a cooled beryllium filter and window. The front end is of a new design and is significantly simpler and less expensive than previous versions. It gives two fans of radiation, each 7 mrad in horizontal aperture. The fan width can be tailored to a specific application with a simple modification to a cooled aperture plate, an improvement that is now the standard for ALS front ends.

Beamline 4.0.1-2

Beamline and undulator construction have been slower than expected, due to problems at the vendor with the monochromator and problems at ALS with the undulator support system. The elliptically polarizing undulator (EPU) has two sets of magnets, and the polarization is changed by phasing diagonally opposite pairs of magnets with respect to the other pair at 90 degrees. The EPU period is 5 cm, and it occupies 2 m of the straight section. The straight section is divided into two parts, with two chicaned vacuum chambers separated in the horizontal plane by 2.5 mrad. The vacuum chambers and chicane magnets have been installed and commissioned. To facilitate installation, the EPU is built on a C-frame support and drive system, and when first tested, it was found to have too great a deflection at minimum gap when driving the phase adjustment. The frame was reanalyzed and, based on the results, strengthening plates were added in several places. The new frame has performed well in extensive testing. The magnetic structure uses shims to position each block, and after two cycles of shimming, the field quality was well within specification. The monochromator has passed all of its qualification tests and has been shipped. Several problems arose during the construction process related to stability, but all were successfully addressed and the system eventually passed a comprehensive set of qualification tests. The monochromator is a version of the Peterson SX-700, having a silicon internally cooled plane premirror, a Glidcop water-cooled grating, and a sagittal focusing refocus mirror. The mechanical system is complex due to the large tuning range required (20–2000 eV) to accommodate the future use of an interchangeable 8-cm-period EPU. The EPU has now been installed in the storage ring and tested at low K. Light has been extracted out of the shield wall and focused onto the entrance slit of the monochromator by a horizontally focusing toroidal mirror, designed to focus on the entrance slit in the vertical direction and at the experimental end chamber in the horizontal direction. The remaining parts of the system are in place and await arrival of the monochromator. The endstation is rotatable, with two separate air bearing tables for individual experiments. Each table can be separated and replaced by systems waiting on other air bearing tables.

Beamline 5.0.2

Beamline 5.0.2 for protein crystallography is powered by a 2.1-T, 37-pole wiggler. The beamline uses a conventional arrangement of a vertically collimating premirror, a constant-exit-height double-crystal monochromator, and a toroidal refocusing mirror. The premirror is silicon and has a double-channel cooling arrangement in which two holes were gun-drilled along the length of the mirror and sealed to waterlines at each end with O rings. The monochromator employs a minichannel finned silicon crystal with internal water cooling. The system worked well from the outset, but it was noticed that at high current the beam moved vertically in the endstation hutch. This was traced to Compton-scattered-radiation heating of the mirror support plate, and was eliminated by fitting the premirror system with a water-cooled guard plate. The beam size at the sample is around 0.5 mm in diameter, with a flux at 12 keV of around 10^{12} photons/s. Typical collection times for complete data sets on average crystals are 2–3 hours, and complete multiple-wavelength anomalous diffraction (MAD) data sets have been taken in under 5 hours. The detector is a 2×2 ADSC CCD-based system. The system is providing world-class performance in terms of throughput as well as in the range and difficulty of structures being solved, including large-unit-cell crystals and microcrystals. In the latter area, structures of crystals down to $30 \times 30 \times 10$ microns have been determined. The permanent mirrors have been designed and ordered, and it is expected that the new premirror and its bending mechanism will be installed in March 1999. This, together with a new refocus mirror, will give a flux increase of 6, and will offer improved spectral resolution over that presently obtained. A small elliptical mirror was also built and tested to see the effectiveness of demagnification for microcrystal diffraction. It was shown that a beam size of 35 μm could be produced with an overall gain in flux density of 7. This mirror does not reduce the resolution of the diffraction pattern as it simply makes the vertical convergence similar to the horizontal convergence.

Beamline 7.0.1

The scanning transmission x-ray microscope (STXM) has been in routine operation on this undulator beamline for two years. Absorption contrast imaging and bulk near-edge absorption spectroscopy (NEXAFS) can be performed in a transmission geometry on submicron regions of thin samples. The instrument development has been described in a recent paper.¹ The major advance of the year has been an improvement in resolution to 67 nm. This resulted from the use of higher-resolution zone plates and by vibration isolation of the microscope support structure. The zone plate has a diameter of 90 μm , a 45-nm outer zone width, and a 30-micron-diameter central stop. It is part of the first production run of the LBNL nanowriter, operated by Erik Anderson and Bruce Hartenek of the Center for X-Ray Optics. The focal length is 1.27 mm, giving a theoretical demagnified image size of the source pinhole of 42 nm. This, convoluted with the diffraction-limited resolution of the zone plate given by the outer zone width, gives the resolution measured. Images are typically acquired with a counting time of 1–5 ms per pixel. NEXAFS spectra are obtained by the acquisition of many images over a selected spectral range of photon energy. Coordinated undulator and monochromator moves between successive images take about 250 ms, during which time a fast mechanical shutter closes to protect radiation-sensitive samples from unnecessary exposure. Due to unwanted motion of the zone plate, the focus moves laterally during photon energy scanning. The images are shifted into registry using a correlation technique, and NEXAFS spectra are extracted from small areas, normalizing against an open area of the image.² The system will be upgraded again over the next year, and we will be focusing on replacement of the zone plate z scan stage so that motion, if any, is reduced during energy scans.

¹Warwick, T., H. Ade, S. Cerasari, J. Denlinger, K. Franck, A. Garcia, S. Hayakawa, A. P. Hitchcock, J. Kikuma, S. Klinger, J. Kortright, G. Meigs, G. Morisson, M. Moronne, S. Myneni, E. Rightor, E. Rotenberg, S. Seal, H.-J. Shin, W.F. Steele, T. Tylicszak, and B.P. Tonner, "A scanning transmission x-ray microscope for materials science spectromicroscopy at the Advanced Light Source," *Rev. Sci. Instr.* **69**, 8 (August 1998), pp. 2964-2973.

²STACKs program; C. Jacobsen, private communication and A.P.Hitchcock, private communication.

Beamline 7.3.1.1

The photoemission electron microscope (PEEM) is now in full operation and has achieved high spatial resolution. It was developed under a DOE Cooperative Research and Development Agreement (CRADA) between the ALS and Joachim Stöhr of IBM. The microscope has electrostatic focusing, and consists of a 30-kV immersion lens objective and two projector lenses, as well as a relay lens used for positioning an image of the objective back focal plane at the position of a set of interchangeable apertures. For flat, reasonably conductive surfaces, the microscope achieves its theoretical minimum resolution of 20 nm. The data rate of the system is good, with images typically taking a few seconds to acquire. This is a result of the highly optimized optical system used to illuminate the sample. This system utilizes a spherical grating to focus at approximately 1:1 in the vertical direction and a 1.2-m-long elliptical mirror to focus in the horizontal direction. This produces a monochromatic focus size of around 30 μm in diameter, with a flux of around 10^{12} photons/s in a 1/1000 bandpass. Magnetic imaging studies have begun, and 50-nm resolution has been achieved using magnetic circular dichroism as a contrast mechanism. The scientific program is centered around studies of magnetic domain structures, antiferromagnetic surfaces, and polymer surfaces. The system is equipped with a comprehensive sample preparation facility, with *in-situ* growth capability. A fully aberration-corrected microscope, which should allow imaging at 2-nm resolution, is in design. Construction of this microscope is expected to start in mid-1999.

Beamline 7.3.3

Beamline 7.3.3 is shared between three programs: an ultrafast diffraction program led by Roger Falcone (University of California, Berkeley) and Phil Heiman, a program to develop ultrafast gating techniques led by Ernie Glover, and an in-house materials diffraction program led by Alastair MacDowell. While synchrotrons have traditionally provided information about the static properties of materials important to the physical and life sciences, they have played only a limited role in studying material dynamics: the time resolution available at synchrotrons (~30 ps) is inadequate for observing the fast (~100 fs) primary events that drive many interesting dynamical processes such as chemical reactions and phase transitions. The ability to perform x-ray spectroscopy with femtosecond time resolution represents an important scientific frontier that will have an impact a number of fields in physics, chemistry, and biology. The goal of performing femtosecond x-ray spectroscopy at the ALS is being pursued via two routes: (1) development of an ultrafast x-ray source and (2) development of ultrafast x-ray detectors. In the work led by the Falcone UCB team, a Ti:Al₂O₃-based laser system produces pulses with a duration of about 100 fs. X rays are detected by an avalanche photodiode (APD) having 5-ns time resolution or a streak camera with a time resolution 1 ps. In one study, x-ray diffraction has been applied to the laser-induced melting of a semiconductor, InSb. Experimental rocking curves are used to evaluate the depth of the melted layer, the strain profile into the crystal and relaxation processes.¹ In a second experiment, NEXAFS is used as a probe of the electronic structure of a silicon foil in transition from solid to liquid to gas.

Development of x-ray detectors faster than 0.5 ps will be extremely challenging; however, recent experiments at LBNL² have demonstrated a physical mechanism upon which a femtosecond x-ray detector can be built. In brief, an intense femtosecond laser pulse is used to modify the continuum into which an x-ray photoelectron is injected, resulting in laser-induced modifications to an x-ray photoelectron energy spectrum (XPS). Modifications to the continuum are twofold. First, the continuum is dressed with optical photons so that an x-ray photoelectron is inclined to absorb or emit these optical photons (thus changing the photoelectron energy). Second, the laser causes an ac stark shift of the continuum level, resulting in an overall shift (to lower energy) of the x-ray photoelectron spectrum. These laser-induced XPS modifications can be used to construct a femtosecond x-ray detector. An ALS x-ray pulse (30–70 ps) and a femtosecond laser pulse illuminate a sample and a photoelectron spectrum is measured. Laser modifications to the XPS result from a narrow slice in time when the femtosecond laser pulse overlaps the long synchrotron x-ray pulse; the laser pulse therefore serves to "optically gate" the x-ray pulse on a femtosecond time scale. By moving the laser pulse through the x-ray pulse in time, fine time structure on the x-ray pulse can be observed, thus permitting ultrafast x-ray spectroscopy. In this approach to developing fast x-ray detectors, time resolution is limited only by the laser pulse duration, which can be as short as 5–10 fs. Experiments are underway to demonstrate optical gating on Beamlines 7.3.3 and 6.3.2 using femtosecond laser systems synchronized to the storage ring.

The diffraction program is centered around two objectives: (1) developing and applying the techniques of x-ray microdiffraction and (2) developing new x-ray user programs in diffraction. The latter usually means that we construct apparatus for demonstration experiments, carry out sets of experiments, and using this data, work with the user group in seeking funds through proposals to funding agencies for dedicated facilities. This has centered around programs in materials chemistry powder diffraction, high-pressure diffraction, protein microcrystal diffraction, and phase contrast tomography. The microdiffraction program is in the process of transferring from Beamline 10.3.2, and a new set of microfocus mirrors and a four-crystal monochromator has been

constructed. This system will be installed in July 1999. The goniometer, detector, and computer control system is under manufacture by Bruker-Siemens and uses a Huber diffractometer.

¹Larsson, J., Z. Chang, E. Judd, R.W. Falcone, P.A. Heimann, H.A. Padmore, P.H. Bucksbaum, M. Murnane, H. Kapteyn, R.W. Lee, and J.S. Wark, *Optics Letters* **22**, 1012 (1997).

²Glover, T.E., R.W. Schoenlein, A.H. Chin, and C.V. Shank, *Phys. Rev. Lett.* **76** (1996), p. 2468.

Beamline 9.3.2

A team led by Malcolm Howells (including Scott Locklin, Eddie Moler, and Zahid Hussain) has constructed an all-reflection Fourier transform spectrometer over the past few years.¹ This system should produce ultrahigh resolution (resolving power of 10^6) and will be used for a variety of gas-phase atomic and molecular science studies over the energy range from 40–120 eV. In the past year, the first results have been obtained and a resolution of 250 μeV has been obtained in the visible region at 1.4 eV photon energy. The theoretical resolution is around 65 μeV . Part of the difference is due to the fact that only 1/2 of the total scan range was used (the resolving power is simply the ratio of the scan length to the wavelength) and the rest is attributed to a combination of Doppler and collisional broadening as well as misalignment. The system is coupled to Beamline 9.3.2 through a branchline after the exit slits, and experiments are now underway with synchrotron radiation to extend the operation to higher photon energies.

¹Moler, E.J., R.M. Duarte, M.R. Howells, Z. Hussain, C. Oh, and J. Spring, “First measurements using the ALS soft x-ray Fourier transform spectrometer” in *Coherent Electron-Beam X-ray Sources and Applications A*, Proc. SPIE **3154**, K. Freund, H.P. Freund, and M.R. Howells, Eds. (Bellingham, 1997).

Beamline 10.3.2

Beamline 10.3.2 was built by the Center for X-Ray Optics and over the past few years had been used as a place to operate a deep-lithography (LIGA) station. With that program now having a home on Beamline 3.3.2, this station was available for other developments.

The ESG has developed a microfocusing system capable of x-ray diffraction and x-ray absorption spectroscopy in the intermediate energy range from 5–12 keV.^{1,2} The x-ray hutch is located 31 m from the source and uses a four-crystal monochromator and a Kirkpatrick-Baez mirror pair to form a monochromatic focus with a spatial resolution of 0.8 μm . The micro x-ray diffraction ($\mu\text{-XRD}$) program has generated significant interest from the thin-film materials community for the quantification of strain in single grains and has spun off into a new program on Beamline 7.3.3. The activities on Beamline 10.3.2 are now centered on an environmental science program, led by Geraldine Lamble, using micro x-ray absorption spectroscopy ($\mu\text{-XAS}$) for local chemical speciation. This has been successful in generating a new user community, and has led to the need for a more permanent, dedicated experimental system. An upgraded beamline has been designed, consisting of a 1:1 relay mirror followed by an x-y slit, a collimating mirror, a four-crystal monochromator, and Kirkpatrick-Baez microfocus optics. This system will have the ability to change resolution by altering the size of the x-y slit, so that users can trade flux for resolution. The sample stage will be replaced with a fast-scan system, and the detector will be replaced with a nine-element system with DSP-based electronics. The new system is planned for commissioning in December 1999.

¹MacDowell, A.A., C.H. Chang, H.A. Padmore, J.R. Patel, and A.C. Thompson, “Grain orientation mapping of passivated aluminum interconnect lines with x-ray micro-diffraction,” in *1998 Spring Meeting of the Materials Research Society*, Applications of Synchrotron Radiation Techniques to Materials Science IV, **524** (1998), pp. 55-58.

²MacDowell, A.A., C.H. Chang, G.M. Lamble, R.S. Celestre, J.R. Patel, and H.A. Padmore, “Progress towards sub-micron hard x-ray imaging using elliptically bent mirrors and its applications,” presented at SPIE conference, San Diego, July 19-24 1998. To be published in SPIE **3449** (1998).